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# Large-Scale Dewatering of Phosphatic Clay Waste From Northern Florida

By Annie G. Smelley and B. J. Scheiner



UNITED STATES DEPARTMENT OF THE INTERIOR



**Report of Investigations 8928**

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**UNITED STATES DEPARTMENT OF THE INTERIOR**  
**Donald Paul Hodel, Secretary**

**BUREAU OF MINES**  
**Robert C. Horton, Director**

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## CONTENTS

|                                           | <u>Page</u> |
|-------------------------------------------|-------------|
| Abstract.....                             | 1           |
| Introduction.....                         | 2           |
| Acknowledgments.....                      | 3           |
| Description of phosphatic clay waste..... | 3           |
| Description of FTU operation.....         | 3           |
| Results and discussion.....               | 3           |
| Consolidation in pit.....                 | 3           |
| Chemical treatment.....                   | 6           |
| Conclusions.....                          | 8           |
| References.....                           | 9           |

## ILLUSTRATIONS

|                                                                                         |   |
|-----------------------------------------------------------------------------------------|---|
| 1. Field test unit (FTU).....                                                           | 4 |
| 2. Flow sequence for FTU.....                                                           | 4 |
| 3. Dependence of time of consolidation to 30 pct solids on depth of water<br>table..... | 6 |

## TABLES

|                                                                                                                             |   |
|-----------------------------------------------------------------------------------------------------------------------------|---|
| 1. Results of FTU test using clay waste treated in pit.....                                                                 | 5 |
| 2. Consolidation of dewatered material in test pit at Suwannee River Mine.....                                              | 5 |
| 3. Laboratory test conditions producing optimum dewatering results.....                                                     | 6 |
| 4. Comparison of effect of lime and $H_2O_2$ in laboratory tests with FTU tests...                                          | 7 |
| 5. Results of measurements on untreated phosphatic clay waste samples.....                                                  | 7 |
| 6. Continuous dewatering tests using phosphatic clay waste pretreated with<br>lime and $H_2O_2$ after 1-h pretreatment..... | 8 |

# UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

|         |                     |         |                         |
|---------|---------------------|---------|-------------------------|
| cP      | centipoise          | lb      | pound                   |
| ft      | foot                | min     | minute                  |
| gal     | gallon              | mL      | milliliter              |
| gal/min | gallon per minute   | μm      | micrometer              |
| g/mL    | gram per milliliter | μmho/cm | micromho per centimeter |
| h       | hour                | pct     | percent                 |
| in      | inch                | ppm     | parts per million       |
| L       | liter               |         |                         |

# LARGE-SCALE DEWATERING OF PHOSPHATIC CLAY WASTE FROM NORTHERN FLORIDA

By Annie G. Smelley<sup>1</sup> and B. J. Scheiner<sup>2</sup>

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## ABSTRACT

The Bureau of Mines is testing a dewatering technique for phosphatic clay waste that will recover a portion of the water lost using conventional waste disposal methods and produce solids suitable for land reclamation. The technique utilizes a flocculant, polyethylene oxide (PEO), that forms strong, stable flocs that can be dewatered on a static screen followed by further dewatering on a rotary screen. In field tests conducted in northern Florida, clay wastes containing a nominal 2.5 pct solids have been consolidated to greater than 20 pct solids. The rate at which PEO-treated material continues to dewater in a mine cut also was monitored. Preliminary results indicate that PEO-treated material will dewater to 30 pct solids in 140 days. Pretreatment of phosphatic clay waste with lime and  $H_2O_2$  was shown to improve the dewatering technique for problem clay wastes.

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## INTRODUCTION

Phosphatic clay waste is one of the principal waste products resulting from the beneficiation of Florida phosphate ore. This waste material, which is comprised of ultrafine particles (70 pct less than 1  $\mu$ m), responds poorly to conventional solid-liquid separation techniques and is currently disposed of by impoundment behind earthen dams.

The overall natural settling rate of phosphatic clay waste is extremely low (1).<sup>3</sup> Generally, impounded clay wastes thicken from a nominal 3 pct solids to 10 pct solids in about 3 months. Densification to about 18 pct solids may require a year, and to reach solids contents of 20 pct or higher may require several years. Data indicate that approximately 90 pct of the water pumped to the settling pond is eventually recovered. The remaining 10 pct of the water lost by storage with the waste clays and by evaporation amounts to about 5 tons for each ton of phosphate concentrate produced (2).

The discharge rate of phosphatic clay waste to settling areas ranges between 20,000 and 80,000 gal/min. To accommodate the waste, impoundments covering 400 to 600 acres, with dam heights ranging from 20 to 40 ft, are required (2-3). At present, in Florida there are approximately 80,000 acres of active and inactive clay settling areas.

To eliminate the use of aboveground disposal, which appears to be the ultimate goal of the State of Florida, new technology is required to process the wastes generated by most of the Florida phosphate mines. To use only mined-out areas for disposal of the clay wastes requires that the solids content of the

material be consolidated from an initial 2 to 6 pct to a final 30 to 38 pct. Generally, at this degree of consolidation, the volume of clay waste mixed with the sand from the phosphate beneficiation sequence would fit into the mine cut and eliminate the need for aboveground storage.

The Bureau of Mines is testing a dewatering technique for phosphatic clay waste that may assist in eliminating storage by impoundment (4-10). A portion of the water now being lost using conventional waste disposal methods also can be recovered, and at the same time the dewatered solids produced may be more suitable for land reclamation. The procedure consists of flocculating the clay waste with a long-chain polymer, polyethylene oxide (PEO). Combining the phosphatic clay waste with PEO forms strong flocs which are tough enough to be dewatered on a screen. In small-scale continuous tests, clay waste was consolidated from a nominal 3 pct solids to 20 pct solids (10). Based upon these results, a field test unit (FTU) was operated at Estech's Silver City Mine, near Bartow, FL. Consolidated phosphatic clay material containing 20 pct solids was produced when feed slurries of 3 pct solids were treated with dosages of PEO as low as 0.7 lb per ton of feed solids (6). Pit and column tests indicated that the PEO-treated material continued to dewater to 30 pct solids in 30 to 90 days, depending on the level of the ground water (6).

This report describes further testing of the PEO dewatering technique at Occidental's Suwannee River Mine in northern Florida. The investigation included optimization of the dewatering sequence and determining the consolidation rate of the dewatered material when placed in a pit that was 9.5 ft below the ground water level.

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<sup>3</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

## ACKNOWLEDGMENTS

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FL. The authors express their appreciation to J. Drummond and F. Sweat of Occidental.

## DESCRIPTION OF PHOSPHATIC CLAY WASTE

The solids from many different phosphatic clay waste samples from Florida were analyzed by X-ray diffraction. The results indicate that the major constituents were apatite, quartz, montmorillonite, and attapulgite, with the ratio of montmorillonite to attapulgite clay

varying over a wide range. Other phosphate minerals such as wavellite and crandallite, in addition to kaolinite, illite, and dolomite, were also found in various concentrations depending on the phosphate deposit.

## DESCRIPTION OF FTU OPERATION

Figure 1 shows the trailer-mounted FTU on location at Occidental's Suwannee River Mine near White Springs, FL. The flow sequence of the operation is shown schematically in figure 2. The equipment included holding tanks for PEO solutions, slime, and makeup water, a mixing tank for preparing PEO solutions, a conditioner for mixing PEO solutions with the clay waste, a static screen, a trommel screen, and various pumps to transport solutions, slurries, and consolidated material. The static screen measured 8 ft wide by 4 ft long with 2.75-in-long and 0.030-in-wide horizontal openings. The trommel, constructed of 10-mesh stainless steel screen with the first 4 ft lined with a 48-mesh screen to prevent loss of solids as the flocculated material flowed into the trommel, was 3 ft in diameter by 12 ft long.

Phosphatic clay waste was pumped to a 5,000-gal holding or feed tank. From there, waste was pumped at 60 to 200 gal/min to a mixer where PEO solution was

added. The resulting mixture was discharged into a trough which overflowed onto the static screen. Released water was removed as underflow as the consolidated flocs moved down the screen by gravity and into the trommel, where the flocs formed a doughlike cylinder (a roll) as water continued to be released. The dewatered material discharged from the trommel was pumped with a positive displacement pump to a pit, where further dewatering was monitored.

A commercially manufactured PEO was used as the flocculant in all the dewatering tests. The polymer, a nonionic water-soluble linear molecule, is composed of repeating units of  $\text{CH}_2\text{-CH}_2\text{-O}$  and has a molecular weight of approximately 5 million. Solutions of PEO at concentrations of 0.25 pct were prepared in 90-gal batches and stored in holding tanks. Water recovered from the static screen underflow was used for polymer solution preparation and for dilution of PEO solutions.

## RESULTS AND DISCUSSION

### CONSOLIDATION IN PIT

The FTU was routinely operated at a fixed rate of 165 gal/min.

"Blue Gumbo Clay," a term used by the industry to describe a clay waste

containing some monovalent exchange ions and unoxidized ions such as sulfide, was present in the Suwannee River phosphate matrix (11). Therefore, it was necessary to pretreat the phosphatic clay waste with hydrated lime,  $\text{Ca(OH)}_2$ , and/or to aerate it to prevent deterioration of the





FIGURE 1. - Field test unit (FTU).

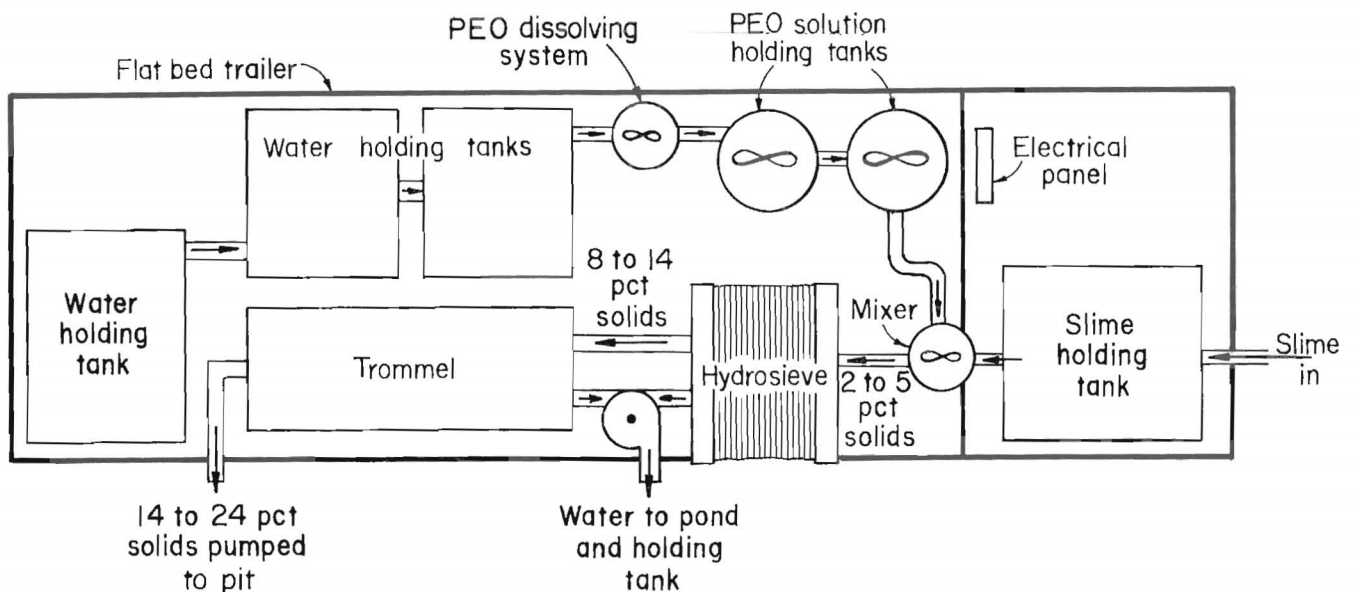


FIGURE 2. - Flow sequence for FTU.

flocs before or during the dewatering sequence (6). To determine the amount of pretreatment required to produce solids of 20 pct or greater, a series of batch tests was conducted in which clay waste was placed in 5,000-gal holding tanks, treated with various amounts of lime, and agitated by pumping the slurry from

the bottom of the tank back to the top for periods of time ranging from 1 to 20 h prior to PEO flocculation. With this treatment, dewatered products of 19 to 26 pct solids could be produced consistently with a lime dosage of 1 to 5 lb/ton and a retention time of 8 to 20 h.

To improve the continuous operation, a pit having a capacity of approximately 200,000 gal was constructed. Clay waste was mixed with lime at a dosage of 5 lb per ton of clay solids and allowed to flow into the pit. Agitation of the clay waste in the pit was accomplished by using four pumps, each pumping at a rate of 120 gal/min. An example of the test results obtained is shown in table 1. The FTU was operated for a 6-h test taking clay waste from one end of the pit while freshly treated clay waste was added to the opposite end of the pit. The PEO dosage ranged from 1.04 to 1.44 lb/ton. The solids content of the discharge from the static screen ranged from 9.3 to 9.9 pct, and that for the trommel was 18.3 to 21.2 pct. Although some solids were lost in the underflows (less than 0.1 pct from the static screen and 0.1 to 0.5 pct from the trommel), they settled quickly in a shallow settling pond, allowing clear water to be recovered from the pond. From 70 to 80 pct of the water contained in the phosphatic clay waste was recovered in the static screen underflow; 10 to 15 pct of the water was recovered in the trommel underflow.

TABLE 1. - Results of FTU test using clay waste treated in pit<sup>1</sup>

| Cumulative time, min | PEO dosage, lb/ton | Solids discharged, pct |         |
|----------------------|--------------------|------------------------|---------|
|                      |                    | Static screen          | Trommel |
| 35                   | 1.04               | 9.3                    | 20.3    |
| 50                   | 1.21               | 9.7                    | 20.3    |
| 95                   | 1.44               | 9.9                    | 21.2    |
| 150                  | 1.29               | 9.6                    | 20.0    |
| 190                  | 1.27               | 9.7                    | 19.4    |
| 230                  | 1.17               | 9.8                    | 19.0    |
| 315                  | 1.16               | 9.9                    | 19.5    |
| 360                  | 1.17               | 9.8                    | 18.3    |

<sup>1</sup>PEO concentration used: 0.25 pct.

Since the trommel discharge solids were somewhat less than the 30 to 38 pct solids necessary for use in land reclamation, it was important to know whether the material continued to dewater. To study the behavior of the dewatered material in a simulated mine cut, a pit approximately 42 by 64 by 11 ft deep was

filled with dewatered clay waste. The ground water level was 9.5 ft above the bottom of the pit. The filling required operation of the FTU at a slime feed rate of 165 gal/min for 141 h and was completed on March 2, 1982. During the filling period, almost 1.3 million gal of clay waste was treated in the FTU and approximately 164,000 gal of dewatered material was deposited in the pit. The FTU was operated for 141 h over a 14-day period, usually during the day shift except when around-the-clock runs of 25 and 31 h were made. The initial solids content of the slime ranged from 1.25 to 4.03 pct. The solids content of the trommel discharge fluctuated during the test period, probably owing to the changing character of the slime coming from the beneficiation plant. The average PEO dosage during the test period was 1.26 lb per ton of clay solids treated. After filling, the pit was sampled periodically at five different locations at 2-ft depth intervals. Data showing the consolidation of the clay waste in the pit as a function of time are presented in table 2. The last sample was obtained 140 days after completion of filling and showed that the material had dewatered from 19 to 30 pct in 140 days.

TABLE 2. - Consolidation of dewatered material in test pit at Suwannee River Mine

| Time, days | Average solids, pct |
|------------|---------------------|
| 0.....     | 19                  |
| 32.....    | 24                  |
| 50.....    | 27                  |
| 64.....    | 28                  |
| 140.....   | 30                  |

From previous tests conducted at Estech (6), it was observed that in a pit 5 ft above the water table, dewatered material reached 30 pct solids in 30 days and 30 pct in 90 days when placed in a column 3 ft below the water table. A comparison of the time required to reach 30 pct solids versus the depth of the water table for the Estech and Occidental experiments is shown in figure 3. A straight line is obtained, indicating that the single most

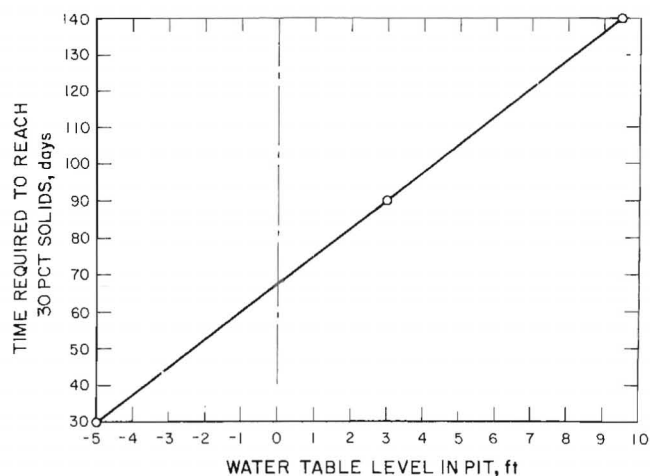


FIGURE 3. - Dependence of time of consolidation to 30 pct solids on depth of water table.

important factor influencing the rate of consolidation in a small pit is the depth of the water table.

#### CHEMICAL TREATMENT

For the Occidental phosphatic clay waste, pretreatment with lime and a holding time of 8 to 20 h, followed by PEO flocculation and dewatering in the FTU, produced a dewatered material generally greater than 20 pct solids. Due to the high rate (60,000 gal/min) of phosphatic clay waste produced at Occidental, a holding time of 8 to 20 h would require a large volume. To reduce the holding time, a series of experiments was conducted to determine if chemical additions or treatments could be substituted for holding time in the dewatering sequence.

Chemicals such as hydrogen peroxide ( $H_2O_2$ ), potassium permanganate ( $KMnO_4$ ), sodium hypochlorite ( $NaOCl$ ), ferric sulfate ( $Fe_2(SO_4)_3$ ), and ozone ( $O_3$ ) were tested, with emphasis being on the use of  $H_2O_2$  since the degradation products are innocuous, i.e., oxygen and water.

Laboratory tests were conducted on samples of phosphatic clay waste that could not be dewatered using the PEO flocculation technique without pretreatment. Aliquots of the wastes were mixed with various proportions of lime and  $H_2O_2$ ; then they were titrated with PEO at specified time intervals. Optimum conditions for several different systems are presented in table 3.

From these results, it is seen that the severity of the problem with such wastes varies over a wide range, and addition of too little or too much  $H_2O_2$  lengthens the required retention time. However, pretreatment with a reagent combination of 1 to 4 lb/ton lime and 0.1 to 0.75 lb/ton  $H_2O_2$  for periods up to 3 h allowed the "problem" wastes to be dewatered to a suitable solids content.

Based on these results, a series of batch tests was conducted on the FTU. Batches of approximately 10,000 gal of phosphatic clay waste received directly from the plant washer were treated with approximately 5 lb/ton lime and various amounts of  $H_2O_2$ . Higher lime dosages were used to ensure adequate additions despite variations in solids content.

TABLE 3. - Laboratory test conditions producing optimum dewatering results

| Sample | Additives, lb/ton |          | Time, h | PEO dosage, lb/ton | Solids, pct |       |
|--------|-------------------|----------|---------|--------------------|-------------|-------|
|        | Lime              | $H_2O_2$ |         |                    | Initial     | Final |
| 1-6... | 0                 | 0        | 3       | 3                  | 7           | (1)   |
| 1..... | 1                 | .4       | 1       | 2.60               | 7.32        | 28.8  |
| 2..... | 2                 | .1       | 1       | 2.33               | 7.31        | 27.1  |
| 3..... | 2                 | .2       | 1       | 2.60               | 7.11        | 28.3  |
| 4..... | 2                 | .75      | 1       | 3.01               | 7.32        | 27.3  |
| 5..... | 4                 | .1       | 1       | 2.24               | 7.49        | 27.5  |
| 6..... | 4                 | .4       | 3       | 2.08               | 7.49        | 29.9  |

<sup>1</sup>No products.

The reagents and clay waste were added to the tanks over a period of about an hour. During this time a composite untreated sample was prepared by taking 1-L samples every 5 min as the tanks filled. The treated waste was sampled when the tanks were full and generally at 45-min intervals thereafter until laboratory tests indicated that the material would dewater. Then the FTU was operated at a feed rate of 165 gal/min using 0.25 pct PEO. The results shown in table 4 support the conclusions from the previous tests. Tests 4 and 7 are examples of too much and too little  $H_2O_2$ , respectively, and too little time.

Since the laboratory tests correlated so well with the FTU tests, an on-site study was made at the end of the campaign to find a set of conditions that would encompass and treat the extreme variations in the clay waste. During this study samples were taken from the plant clay waste line over a period of several days, and the following properties were measured: conductivity, pH, viscosity, density, solids content, and degree of dewatering. Typical data from this series of experiments are presented in table 5.

TABLE 4. - Comparison of effect of lime and  $H_2O_2$  in laboratory tests with FTU tests

| Test | Initial solids, pct | Additives, lb/ton |                 | Laboratory     |                    |                     | FTU            |                    |                               |
|------|---------------------|-------------------|-----------------|----------------|--------------------|---------------------|----------------|--------------------|-------------------------------|
|      |                     | Lime dosage       | $H_2O_2$ dosage | Mixing time, h | PEO dosage, lb/ton | Product, pct solids | Mixing time, h | PEO dosage, lb/ton | Trommel discharge, pct solids |
| 1... | 1.82                | 6.7               | 1.3             | 2.25           | 1.92               | 21.4                | 2.75           | 1.18               | 20.3                          |
| 2... | 2.57                | 4.8               | .7              | .75            | 1.56               | 27.5                | 1.00           | .90                | 22.3                          |
| 3... | 2.94                | 4.2               | .4              | 1.50           | 1.53               | 30.5                | 1.75           | 1.10               | 31.7                          |
| 4... | 2.71                | 4.5               | .7              | 0              | 2.95               | 13.5                | .50            | 1.34               | 11.0                          |
| 5... | 3.43                | 3.6               | .5              | 2.25           | 2.04               | ( <sup>1</sup> )    | 2.50           | 1.63               | 27.1                          |
| 6... | 3.74                | 3.3               | .65             | 2.25           | 1.60               | 27.4                | 2.50           | 1.11               | 27.3                          |
| 7... | 4.65                | 2.6               | .13             | 1.50           | 3.23               | ( <sup>1</sup> )    | 2.00           | 2.23               | 13.9                          |

<sup>1</sup>No products.

TABLE 5. - Results of measurements on untreated phosphatic clay waste samples

| Test | Conductivity, $\mu$ mho/cm | pH   | Dissolved oxygen, ppm | Viscosity, cP | Density, g/mL | Initial solids, pct | PEO dosage, lb/ton | Water removed, mL <sup>1</sup> |
|------|----------------------------|------|-----------------------|---------------|---------------|---------------------|--------------------|--------------------------------|
| 1... | 335                        | 7.12 | 4.7                   | 22.4          | 1.0098        | 2.27                | 17.45              | 70                             |
| 2... | 325                        | 7.40 | 4.1                   | 87.7          | 1.0204        | 3.82                | 12.83              | 65                             |
| 3... | 395                        | 6.42 | 4.4                   | 10.3          | 1.02195       | 4.12                | 7.72               | 122                            |
| 4... | 325                        | 6.46 | NA                    | NA            | NA            | NA                  | NA                 | 35                             |
| 5... | 382                        | 6.18 | 4.3                   | 16.8          | 1.0284        | 4.88                | 8.47               | 86                             |
| 6... | 388                        | 6.20 | 5.6                   | NA            | NA            | 4.01                | .51                | 89                             |
| 7... | 405                        | 6.80 | 5.3                   | 11.4          | 1.0121        | 2.28                | 5.09               | 160                            |
| 8... | 445                        | 7.10 | 6.1                   | 4.3           | .9977         | .18                 | 13.92              | 177                            |
| 9... | 408                        | 6.76 | 3.9                   | 17.8          | 1.01235       | 2.39                | 17.15              | 113                            |
| 10.. | 400                        | 6.70 | NA                    | 10.5          | 1.0195        | 3.48                | 6.93               | 186                            |
| 11.. | 428                        | 6.92 | 1.4                   | 19.7          | 1.0220        | 4.05                | 12.08              | 70                             |
| 12.. | 420                        | 7.10 | 4.7                   | 5.6           | 1.0126        | 2.42                | 8.16               | 166                            |
| 13.. | 320                        | 6.76 | 4.1                   | 32.5          | 1.0244        | 4.45                | 6.17               | 100                            |
| 14.. | 348                        | 6.52 | 6.7                   | 9.4           | 1.0043        | 1.11                | 7.40               | 196                            |

NA Not available.

<sup>1</sup>Water recovered from a 200-mL sample of clay waste.

TABLE 6. - Continuous dewatering tests using phosphatic clay waste pretreated with lime and H<sub>2</sub>O<sub>2</sub> after 1-h pretreatment

| Test  | Running time, h       | Feed rate, gal/min | Initial solids, pct |             | Additive dosage, lb/ton |                               |
|-------|-----------------------|--------------------|---------------------|-------------|-------------------------|-------------------------------|
|       |                       |                    |                     |             | Lime                    | H <sub>2</sub> O <sub>2</sub> |
| 1.... | 1.25                  | 165                | 2.28                |             | 6.2                     | 0.65                          |
| 2.... | 4.17                  | 100                | 3.58                |             | 4.3                     | .38                           |
| 3.... | 2.00                  | 100                | 2.63                |             | 5.8                     | .52                           |
| 4.... | 1.75                  | 100                | 2.12                |             | 7.2                     | .65                           |
| 5.... | 3.50                  | 100                | 3.13                |             | 4.9                     | .44                           |
|       | Pre-treatment time, h | PEO dosage, lb/ton | Static screen       |             | Trommel                 |                               |
|       |                       |                    | Underflows, pct     | Solids, pct | Underflows, pct         | Solids, pct                   |
| 1.... | 0.8                   | 1.84               | 0.04                | 7.97        | 0.21                    | 19.9                          |
| 2.... | 1.3                   | 1.71               | .11                 | 13.65       | .22                     | 25.7                          |
| 3.... | 1.3                   | 1.73               | .34                 | 5.13        | .59                     | 11.0                          |
| 4.... | 1.3                   | 1.62               | .20                 | 4.72        | .53                     | 11.2                          |
| 5.... | 1.3                   | 1.25               | .21                 | 11.06       | .18                     | 22.0                          |

Samples were subjected to pretreatment with various chemicals such as H<sub>2</sub>O<sub>2</sub>, KMnO<sub>4</sub>, lime, guar, and chemical polymers of high molecular weight. This pretreatment was evaluated after rather short reaction times, no longer than 4.5 h. The variation in clay waste samples was so great that no prescribed dosage of any of the reagents tested worked for all of the samples. However, it appeared that pretreatment with lime and H<sub>2</sub>O<sub>2</sub> at dosages of 5 and 0.5 lb/ton, respectively, for 1 h before flocculation with PEO would allow sufficient dewatering and thus processing on the FTU.

To test the validity of this conclusion, the FTU was operated continuously under these conditions on 5 separate days by using the two 5,000-gal tanks to achieve the approximate pretreatment time. Lime and H<sub>2</sub>O<sub>2</sub> were added to the phosphatic clay waste as it entered the tanks at a flow rate equal to that used in the FTU operation. With the exception of the first test, the feed rate to the tanks was maintained at 100 gal/min. The results presented in table 6 indicate that pretreatment with dosages of 4 to 5 lb lime and 0.4 lb H<sub>2</sub>O<sub>2</sub> for 1.3 h indeed is equivalent to that described earlier using the pit and 8 to 20 h pretreatment time.

#### CONCLUSIONS

Phosphatic clay wastes from northern Florida were successfully dewatered in an FTU at the rate of 165 gal/min. It was determined that lime addition followed by a holding time of 8 to 20 h was required prior to PEO flocculation if 18 to 21 pct solids were to be produced routinely in the FTU. Also it was demonstrated that the addition of 4 to 5 lb/ton lime and 0.4 lb/ton H<sub>2</sub>O<sub>2</sub> prior to PEO flocculation would reduce the required holding time to 1.3 h.

PEO dewatered material continued to consolidate when placed in a pit and reached 30 pct solids in 140 days. A comparison of the data obtained from this investigation with data obtained from a previous FTU operation indicates that the water table of the pit is a major factor in determining the rate at which dewatered material from the FTU will reach 30 pct solids.

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